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MINERAL FIBRE FELT

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(57) Claim

1. Felt composed of fibres of a glass material, the fibres being produced by passing the material in the molten state through openings disposed on the periphery of a centrifuge, wherein the mass of the fibres that have an apparent diameter greater than 40 micrometers is at most 1% of the total mass of the fibres composing the felt.

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Complete Specification for the Invention entitled: MINERAL FIBRE FELT.

The following statement is a full description of this invention, including the best method of performing it known to me:—

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This invention relates to mineral fibre felts having improved properties.

More precisely, the invention relates to felts in which the fibres, of the type of glass fibres or the like, are produced by passage of the material of which they are constituted through a centrifuge serving as bushing. The felts prepared by this technique have a certain number of properties in common by which they may be distinguished from felts in which the fibres have been prepared by other techniques. It may also be noted that due to the quality required and the operating costs, a very important proportion of the production of felts for thermal and acoustic insulation makes use of a technique of fibre formation of this type.

The felts obtained hitherto are on the whole quite satisfactory in their properties and with suitable choice of the operating conditions it is possible to obtain fine, long fibres with only a low non-fibrous content. These fibres are suitable for the production of felts having advantageous insulating qualities and high mechanical strength, which is indispensable for their use. These various properties will be examined in detail below,

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together with the relationship between the characteristics of the fibres and the quality of the felts. Improvements in these products are nevertheless always desirable and it is this which the invention proposes to provide.

In particular, the invention has the object of providing felts with improved properties but obtained at production costs substantially the same as those required for felts produced prior to this invention.

It is important to emphasize the question of production costs. Other techniques for the production of fibres to be used for insulating felts are known and some of these techniques may result in products having interesting properties but the production costs generally are very substantially higher than those of the technique according to this invention.

It is known, for example, to produce fibres by the technique of mechanical attenuation. These continuous fibres, known as "textiles", may subsequently be cut up into fragments of the required length and distributed at random over a receiver to form a felt. Felts in which the fibres have a very high degree of homogeneity may be obtained by this type of technique. It is this homogeneity to which may be attributed the fact that the felts obtained are of high quality even when the fibres are not extremely fine. This method of fibre production and the operations for forming the felts, however, entail very high production costs.

Another type of technique, known as "aerocor", which

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is also used for the production of high quality fibres, consists of introducing fine rods of solid material into a current of hot gas flowing at high speed. The rods soften under the effect of the heat and are attenuated into long, fine fibres if the conditions have been suitably chosen. This technique is also much more costly than that considered according to the invention.

The felts according to the invention consist of fibres produced by a technique of centrifugation. In this technique, the material which is to form the fibres is introduced into a centrifugal device the peripheral wall of which is perforated by numerous orifices. Under the effect of centrifugation, the material is projected through these orifices in the form of fine filaments which become attenuated. Preferably, a gas current at high temperature and high speed flowing along the peripheral wall of the centrifuge participates in the attenuation of the filament into fine fibres.

High quality fibres may be produced in great abundance by this type of technique and consequently at relatively low cost.

The felts obtained by these techniques generally have quite specific characteristics which enable them to be distinguished from felts prepared by any other method. Thus, as will be seen below, the fibres constituting the felts according to this invention have a good regularity of diameter whereas fibres obtained by mechanical attenuation have a strictly constant diameter. The diameter of

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the fibres produced by mechanical attenuation is also normally larger than the average diameter of the fibres of the felts according to the invention. Moreover, the method of chopping up so called "textile" fibres imposes a strictly specified length on the cut fibres.

Characteristic differences may also be shown between the fibres according to the invention and the fibres produced by gaseous attenuation mentioned above. One important difference is associated with the presence of non-fibrous particles resulting from defects appearing at the time of formation of the fibres. These particles are substantially thicker than the average diameter of the fibres.

In the aerocor type of attenuation techniques, the non-fibrous particles are normally present in large quantities and most often in the form of hooks whereas any non-fibrous particles obtained in the techniques of fibre formation by centrifugation employed for production of the felts according to the invention are always present only in small proportions and most frequently in the form of agglomerated fibres.

The presence of non-fibrous particles in felts is undesirable. Each of these particles constitutes a considerable mass of material compared with that of a fibre. In relation to its mass, a non-fibrous particle contributes very much less to the insulating properties than a normal fibre. In other words, for a given mass of fibrous material, a felt which is free from non-fibrous particles has better insulating properties than a felt containing

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such non-fibrous particles.

The presence of non-fibrous particles is also normally accompanied by a great lack of homogeneity in the characteristics of the fibres as a whole, which is another unfavourable factor for the quality of the felt.

For these reasons, the absence of non-fibrous particles or at least the presence of only a very low proportion thereof is particularly desirable whatever the method of formation of fibres under consideration. The techniques of formation by centrifugation in which the centrifuge serves as bushing are generally known to result in a very low non-fibrous content. The invention particularly relates to felts in which the non-fibrous content is even lower.

In the text of this application, the term "non-fibrous" is used to define any particles having dimensions substantially greater than the average diameter of the fibres. These non-fibrous particles are also characterised by lacking in slenderness, that is to say the ratio of their length to their diameter is low.

In practice, any particle having a diameter greater than 10 times the average diameter of the fibres may be regarded as non-fibrous.

The proportion of non-fibrous particles in the products according to the invention is less than 1% and preferably less than 0.80% by weight.

Also according to this invention, the proportion of non-fibrous particles having dimensions equal to or greater

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than 20 times the average diameter is less than 0.30% by weight of the whole felt.

The average diameter varies according to the nature of the products prepared so that the dimension of particles termed "non-fibrous" also varies. For felts intended to be used for insulation and prepared by techniques such as those envisaged by the invention, the diameter of the fibres varies approximately from 1 to 20 micrometers and for the sake of simplicity, any particle having a diameter greater than 40 micrometers may be regarded as non-fibrous.

According to this definition, the felts according to the invention are distinguished from felts previously obtained by similar techniques in that they contain at the most 1% by weight of particles having a diameter greater than 40 micrometers. This content is preferably less than 0.80%.

The method of classification of the fibres and particles is described in detail below with reference to examples.

Since the measurement is carried out by weight and the non-fibrous particles are by nature heavier than fibres, the actual number of non-fibrous particles is very low.

It may also be noted that, in the products according to the invention, the non-fibrous particles are relatively small compared with those found in products produced by other techniques. A granularmetric study shows that the percentage by weight of particles having a dimension greater than 80 micrometers is less than 0.3%.

The products according to this invention contain virtually no particles having a diameter greater than 100 micrometers.

As already indicated above, the absence of non-fibrous particles is normally associated with great homogeneity in the characteristics of the fibres. This homogeneity may be ascertained, for example, from the variations in the diameter of the fibres. For example, the standard deviation of the fibre diameters, that is to say, the difference between the extreme diameters within the range containing 68% of all the fibres, may be measured on the histogram of a sample of felt.

The co-efficient of variation, that is to say, the ratio of the standard deviation to the average diameter in the sample under consideration, is a measure of the dispersion of the characteristics of the fibres.

In this respect also the felts according to the invention compare favourably with felts previously known. The co-efficient of variation is less than 0.85 and preferably less than 0.60 and may even be as low as 0.5. In other words, the histogram of the fibres in the felts according to the invention is very narrow.

Visual observation of the fibres of the felts according to the invention reveals a regularity of structure substantially better than that of fibres of the felts traditionally prepared by similar techniques of centrifugation. The surface of the fibres appears to be smoother, the cross section of the fibres more constant, deformed fibres, basically consisting of fibres stuck together, are

less numerous etc. It should be understood that these differences are difficult to quantify but they are nevertheless a useful confirmation of the advantageous structural characteristics of fibres constituting the felts according to the invention.

The felts according to the invention are also distinguished by the length of their constituent fibres.

Direct measurement of the length of fibres is an operation entailing great difficulties. Handling of the fibres causes them to break and this constitutes a source of error. Moreover, only statistical data are significant, and this requires a large number of measurements.

To overcome these difficulties, specific, well reproducible methods have been developed in the art.

An indirect method of measuring the length of fibres consists of measuring the sedimentation volume of a suspension of fibres in water. Systemmatic studies carried out on this type of measurement show that for a sample of given mass, the sedimentation volume increases with the length of the constituent fibres. It is understood that since deposition of the fibres takes place at random, entanglement of the fibres will be all the "looser" the greater the length of the fibres.

The sedimentation volume is also a function of the diameter of the fibres. The finer the fibres and therefore the greater their number for a given mass, the larger will be this volume.

Within limited ranges of values, the volume may be considered to be approximately proportional to the length

and inversely proportional to the diameter of the fibres.

This method is generally known by the English term of "bulking". It is a convenient method enabling relative determinations to be obtained for the whole mass of fibres in the sample studied.

The measurements are carried out on fibres whose length has previously been reduced by limited crushing under clearly specified conditions. This preliminary crushing reduces the fibres to several millimetres but this length always remains a function of the initial length.

The measurements of sedimentation height or volume are compared with those of fibres whose initial characteristics are known.

Due to the operation of crushing, the method of bulking is also a means of ascertaining the mechanical resistance of the fibres. It is obvious that fragile fibres, even if initially long, would provide relatively low sedimentation volumes after crushing. This method of measurement provides an overall indication of the two properties, length and resistance.

The substantial increase in sedimentation volume observed in felts according to the invention is necessarily associated with an improvement in the length and resistance of the fibres.

The quality of the fibres constituting the felts according to the invention has been considered above and it is obvious that the improvements in the fibres also affect the insulating and mechanical properties of the felts.

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Comparisons with known products are difficult to establish due to the large number of parameters which contribute to these properties. Insulating felts are traditionally classified commercially according to their thermal resistance R, which is a function of the fineness of the fibres F and the mass of fibres per unit surface area, or "weight" m but also of the thickness of the felt and the qualities of the fibres, as indicated above.

The fineness of fibres is conventionally ascertained by an overall measurement known as "micronaire". The micronaire is determined by the rate of air flow through the sample of fibres of a given mass (for example 5 g). The sample is placed in a compartment situated on a circuit in which air circulates under a specified pressure. The micronaire is measured by the rate of air flow through the sample.

It is also found that for a given thickness of felt, a given fineness of fibres and a given thermal resistance, the felts according to this invention are lighter in weight. In other words, a smaller mass of fibres is sufficient to produce the same insulating effect.

The lower mass per unit area of the felts according to the invention is accompanied by greater regularity of the fibres, absence of non-fibrous constituents and more homogeneous distribution of the fibres within the felt.

For a significant definition of the mass per unit area or weight of the felts, it is necessary to specify the thermal resistance of the felt R and the thickness e. These factors are related by the following expresseions:

$$m/R = m/e \times \lambda$$

$$m/R = m/e\ (A + B\rho + C/\rho)$$

$$m/R = \rho A + B\rho^2 + C$$

In these expressions, λ is the thermal conductivity and ρ the mass per unit volume of the felt.

Since the felts under consideration have a very low mass per unit volume ρ, the factor $B\rho^2$ may be neglected as a first approximation:

$$m/R = \rho A + C$$

When m is expressed in kg/m^2, R in $m^2 : K/w$ at 297^oK and ρ in kg/m^3, the felts according to the invention conform to the condition:

$$m/R \leqslant 0.026\ \rho + 0.2$$

For example, if a felt according to the invention has a mass per unit volume of 10 kg/m^3 and the fibres have a fineness of 3 (under 5 g) the weight required for a thermal resistance of 2 $m^2 . {}^oK/w$ may be of the order of or less than 920 g/m^2.

The advantages of being able to produce lighter weight insulating felts are obvious. The quantity of felt produced from a given mass of fibres is greater and the production costs are therefore less.

The mechanical properties of the felts according to the invention are also substantially improved, particularly the tensile strength.

The so called "ring" tensile strength is measured according to standard ASTM-C-681.76. According to this standard, rings of specified dimensions are cut out of the felt. These rings are placed on two cylindrical

traction bars, where they are subjected to forces of increasing magnitude. The force exerted at rupture is measured. To obtain comparable results, the force exerted is related to the weight of the sample.

The felts according to the invention bonded with a phenolic binder representing at the most 4.5% by weight of the final product preferably have a tensile strength at least equal to 200 gf/g in the longitudinal direction, that is to say, when the rings are cut out so that their length extends in the direction of the felt.

The improvement in tensile strength compared with that of conventional felts may also be attributed to the presence of longer, more regular and more evenly distributed fibres. It will be understood in particular that longer fibres improve the network structure of the felt. It is also obvious that if fibres have a more regular structure so that one may assume that individually they have a greater resistance (measurements carried out on individual fibres but in too small a number to be representative of all the fibres of the felt have shown an increase in the tensile strength), the resistance of the felt will also be improved.

Since the felts according to the invention have improved mechanical properties, they may easily be subjected to considerable compresssion. For storage and transport, it is in fact necessary to compress such bulky, light-weight products. In order that the felts may subsequently recover their insulating properties, they must be able to recover their thickness, even after three months under compression. The felts according to the invention are

capable of satisfactorily withstanding compression rates equal to or greater than 5 and in certain cases as much as 7 or 8, showing that the regular fibres of which they are composed are capable of undergoing considerable deformation without rupture.

The mechanical qualities of the fibres constituting the felts according to the invention are also manifested indirectly by the dust content of these felts.

By "dust" is meant any particles not fixed to the felt and capable of being detached from the felt without great effort.

The presence of dust in the felt is due mainly to the break up of insufficiently resistant fibres at the time of packaging of the felt (cutting, rolling up, compression, etc).

The conditions of measurement are described in the examples.

These measurements show significantly a very low dust content for the products according to this invention. The dust content is less than 0.1 mg/m^2.

Apart from the fact that the absence of dust is an additional indication of the quality of the fibres, it also constitutes an advantage to the user. For this reason also, a product free from dust or emitting little dust is always preferred.

The regularity of the fibres in the felts according to the invention is also apparent in the manner in which they are distributed inside the felt. It is important that the felt should be as homogeneous as possible. In

order that insulation may be effective, the felt should be free from any zones in which the fibres are less abundant.

The even distribution of fibres may manifest itself in particular by the resistance of the felt to the circulation of air. It will be seen in the examples that the felts according to the invention afford advantages also in this respect. In particular, for a density of 10 kg/m^3, the resistance measured according to the standard ISO-DIS-4638 is greater than 4 Rayl/cm; for a density of 20 kg/m^3 it is greater than 10 Rayl/cm.

Production of the fibres and felts having the properties indicated above is carried out according to this invention by a technique comprising centrifugation. More precisely, in the techniques provided, the material from which the fibres are to be formed is introduced in the molten state into a centrifuge having its periphery perforated by numerous orifices. Under the effect of the centrifugal force, the material passes through these orifices and is projected from the centrifuge in the form of filaments.

In this preferred embodiment, an intense current of hot gas travels along the wall of the centrifuge from which the filaments are projected. This hot gas carries the filaments along with it and attenuates them. This technique is advantageous in particular because it enables felts of high quality to be obtained at low cost.

This method of fibre formation has been the object of numerous publications, in particular French Patent Application published under the number 2,443,436.

One means of obtaining fibres and felts having the properties indicated above by this type of technique consists of maintaining the centrifuge at a peripheral speed above 50 m/s while centrifugation is carried out. This speed may be obtained with centrifuges of different diameters but for producing fibres in economic quantities it is preferable to operate with centrifuges having a diameter above 500 mm.

The causes for the improvement in the fibres and felts have not been clearly established. It may be assumed that for various reasons, the fibres in the course of their formation have less tendency to collide before solidifying. The fibres are therefore in general longer and their properties improved. It is also possible that under the conditions maintained, the forces acting on the fibres are more regular and attenuation therefore develops more progressively with less rupture of the filaments or at least more delayed rupture.

Whatever the reasons, the quality of the products is found to be substantially improved, as the examples below will demonstrate.

The results are compared with those obtained with two commercially available products, referred to below as A and B. These two products are prepared, like the felts according to this invention, by centrifugation of the material forming the fibres through the orifices of a centrifuge and attenuation by a gas current.

The results obtained for the factors investigated are given below.

1. Determination of the proportion of non-fibrous constituents

The sample analysed is first freed from the binder contained in it. The sample is subjected to limited crushing with a view to dissociating the non-fibrous constitutents from the fibres to which they are normally attached.

The fibres are then separated from the non-fibrous constituents by passing them with water over a separating column through which an ascending stream of water flows.

The non-fibrous particles are recovered at the bottom of the column. They are dried and passed through a row of vibrating sieves which retain the non-fibrous particles larger than, respectively, 100, 80 and 40 micrometers.

The results, expressed in percentages by weight of the initial sample, are as follows:

non-fibrous consitutuent	A	B	Invention
$\phi > 40 \times 10^{-6}$m	1.7	1.9	0.63
$\phi > 80 \times 10^{-6}$m	1.3	1.6	0.15
$\phi > 100 \times 10^{-6}$m	0.3	1.3	0.05

The felts according to the invention are distinguished by their very low non-fibrous content as well as by the small dimensions of the residual non-fibrous particles.

The systemmatic errors introduced in particular by the method of separation of the fibres from non-fibrous constituents are negligible.

2. Histogram of the diameters of the fibres

Counts are made on the samples of felts from Example 1. The corresponding histogram the average diameter, the standard deviation fand the co-efficient of variation are established from these counts as a function of the diameter measured for each sample of felt.

In these measurements of diameter, the non-fibrous particles are not taken into consideration.

The results are as follows:

	A	B	Invention
Average diameter (10^{-6}m)	5.3	2.9	4.5
Standard Deviation (10^{-6}m)	3.4	2.7	2.5
Coefficient of variation	0.64	0.93	0.5

The low figure for the standard deviation in the case of the invention is particularly remarkable in view of the fact that the fibres of the sample are on average less fine than those of product B. This corresponds to a very narrow histogram. In other words, the fibres of the felts according to this invention have a relatively constant diameter.

3. Estimation of the length of fibres (bulking)

The samples of 5 g of fibres which have been freed from binder by their passage through a furnace at 450°C are crushed for 10 seconds in 500 cc of distilled water.

After crushing, the water and fibres together are

transferred to a graduated 1,000 cc cylinder and left to sediment for 5 minutes (sedimentation virtually complete).

At the end of sedimentation, the upper level of the column of fibres is measured in millimetres. The measurements are repeated at least 3 times and the average value taken.

The results of these measurements are shown in the table below:

	A	B	Invention
Height after 3 min (mm)	41	128	150
Height after 5 min (mm)	39	127	149

The above table shows that the products according to the invention are formed from substantially longer and/or more resistant fibres than those of the analogous felts A and B.

4. <u>Mass per unit surface area</u>

The weight or mass per unit surface area of the various felts and their thermal resistance are shown in the table below:

	A	B	Invention
m g/m^2	1000	1350	915
R $m^2.^\circ C/W$	2	2	2
m/R	500	675	457

The felts according to the invention require less fibres for the same insulating quality.

5. <u>Tensile strength (ASTM-C-681.76)</u>

The samples are punched out in the form of rings. Their dimensions are as follows:

Total length : 122 mm,

Interaxial length : 46 mm,

Internal radius : 12.5 mm,

External radius : 33 mm.

The sample is weighed and then placed on a test machine comprising two cylindrical rods 25 mm in diameter.

The velocity of the movable rod carrying the ring is 200 mm/min.

The force at the time of rupture is measured and the ratio of this force to the mass of the sample calculated.

The measurements are carried out first on samples cut out in the longitudinal direction of the felt and then samples cut out in the transverse direction.

The results are shown below, expressed in gf/g.

	A	B	Invention
Longitudinal resistance	164	148	214
Transverse resistance	159	136	183

The resistance of the felts according to the invention is found to be superior to those of comparison felts in

both directions.

6. Dust content

The dust content is measured on samples measuring 300 x 400 mm. These samples are suspended perpendicularly to a vibrator inside a cabin.

The frequency of the vibrator is 50 Hz and its amplitude 2.5 mm. The vibration is maintained for two hours.

The particles which become detached from the sample are collected at the bottom of the cabin. These particles are weighed and their mass is related to the exposed cross sectional surface of the felt.

In this measurement, only the surface of cross sections passing through the thickness of the felt is taken into account. In fact, virtually the total quantity of particles which become detached are derived from these surfaces, which have been subjected to the most severe stresses, particularly at the time of cutting.

The upper and lower surfaces of the felt allow virtually no dust to pass through since they have been made smooth by the treatment of the felt in the stove.

Felt A is found to have a dust emission of 0.16 g/m^2 of cross section. The corresponding value for the felt according to the invention is only 0.07 g/m^2.

7. Permeability to air

Determination of this permeability, which reflects the evenness of distribution of the fibres, is measured according to the standard ISO-DIS-4638.

According to this standard, a stream of air is passed through a sample having specified characteristics. The pressure drop between the two exposed surfaces of the sample is a measure of its permeability.

Measurements were carried out on products having two distinct densities (10 and 20 kg/m^3). The results are summarised in the table below: They are expressed in CGS units of Rayl/cm.

kg/m^3	A	B	Invention
10	3	3	4.8
20	8	7.5	12

The high performances of the felts according to this invention are confirmed by a simple observation. The fibres are arranged in a clearly stratified form and no zone having a reduced fibre content appears.

Under these conditions and bearing in mind the quality of the fibres indicated above (regularity, absence of non-fibrous constituents, etc), the best resistance to air flow would be consistent with the other results indicated, in particular the improvement in thermal resistance for a given weight (or, what is equivalent, the same thermal resistance is obtained with less weight).

3. Resistance to compression

Light-weight felts, thermal resistance 2 m$^{2\circ}$K/W, having a nominal thickness guaranteed to the user of 90mm

were packaged in the form of rolls in which the thickness of the felt was only a fraction of the nominal thickness.

The maximum compression ratio (ratio of nominal thickness to thickness in the compressed state) which still enables the product to recover its nominal thickness after four months under compression was determined for the products according to the invention and for products A and B.

The following results were obtained:

	A	B	Invention
compression ratio	4	4	5

The results show that the felts according to the invention have a higher resistance to compression. The lower the mass per unit area or weight of the product according to the invention, the better these results, as already indicated previously.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. Felt composed of fibres of a glass material, the fibres being produced by passing the material in the molten state through openings disposed on the periphery of a centrifuge, wherein the mass of the fibres that have an apparent diameter greater than 40 micrometers is at most 1% of the total mass of the fibres composing the felt.

2. Felt as claimed in claim 1 in which the mass of the fibres that have an apparent diameter greater than 80 micrometres is at the most 0.30% of the total mass of the fibres composing the felt.

3. Felt as claimed in claim 1 or claim 2 in which the mass of the fibres that have an apparent diameter which is more than 20 times the mean diameter of the fibres constitutes less than 0.30% of the total mass of the fibres composing the felt.

4. Felt as claimed in any one of the foregoing claims, in which the variation coefficient for the diameter of the fibres is less than 0.65%.

5. Felt as claimed in any one of the foregoing claims, in which the ratio of the grams per square metre, or mass per unit area m/R, expressed in kg/m^2, is to the thermal resistance at 297 K, expressed in $m^2.K/W$, is at most equal to 0.026ρ, ρ being the density of the felt in kg/m^3.

6. Felt as claimed in any one of the foregoing claims, in which the proportion of dust released by a sample subjected to the test as hereinbefore defined is less than 0.1 mg/m^2 edge area.

7. Felt composed of fibres of glass material substantially as hereinbefore described with reference to any one of the foregoing examples apart from the comparative examples.

Dated this 29th day of April, 1988
ISOVER SAINT-GOBAIN
By their Patent Attorneys:
CLEMENT HACK & CO.
Fellows Institute of Patent Attorneys of Australia.